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[CHD 1: Inorganic Chemicals]



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Indian Standard

**SPECIFICATION FOR ROCK PHOSPHATE
FOR CHEMICAL INDUSTRIES**

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR ROCK PHOSPHATE FOR CHEMICAL INDUSTRIES

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Indian Standard

SPECIFICATION FOR ROCK PHOSPHATE FOR CHEMICAL INDUSTRIES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 March 1985, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 The naturally occurring rock phosphate contains several constituents, the principal one being the calcium phosphate. For a long time there were no known sources of rock phosphate in India and the mineral was being imported. It is only recently that huge deposits of this mineral have been found in Mussoorie (Uttar Pradesh) and in the Jhamarkotla mines in Udaipur (Rajasthan). More recently deposits have also been found in Lalitpur (Uttar Pradesh); Jhabua and Hirapur (Madhya Pradesh); Cuddapah (Andhra Pradesh); and Purulia (West Bengal).

0.3 This standard covers rock phosphate as a raw material for the manufacture of elemental phosphorus (Type 1), and phosphoric acid (Type 2) only.

0.3.1 This standard does not cover rock phosphate for use by fertilizer industry for which a separate Indian Standard is under preparation.

0.4 The specification for type 1 rock phosphate for use in the manufacture of elemental phosphorus is based on the furnaces in use at present by manufacturers of elemental phosphorus. However, the Committee making its observations on this specification, emphasized that rock phosphate even with 23 percent phosphates (P_2O_5) and 15 percent silica can be used for the manufacture of elemental phosphorus, if furnace designs are modified accordingly. It was recorded that the indigenous manufacturers should use local sources to the maximum possible extent. The specification for type 1 would be reviewed and revised after three years when industry has examined this possibility further.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes requirements and the methods of sampling and test for rock phosphate used for manufacture of elemental phosphorus, and phosphoric acid (*see* also 0.3.1).

2. TYPES

2.1 The material shall be of following two types:

Type 1 — for use in the manufacture of elemental phosphorus (thermal process); and

Type 2 — for use in the manufacture of phosphoric acid.

NOTE — Type 2 of the material is essentially meant for phosphoric acid for the manufacture of chemicals.

3. REQUIREMENTS

3.1 **Particle Size** — Particle size of the material shall be as agreed to between the purchaser and the supplier.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 5 of Table 1.

TABLE 1 REQUIREMENTS FOR ROCK PHOSPHATE

Sl No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST, REF TO CL No. IN APPEN- DIX A
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	Total phosphates (as P_2O_5), percent by mass, <i>Min</i>	30.0	32.0	A-3
ii)	Silica (as SiO_2), percent by mass, <i>Max</i>	10.0	5.0	A-4
iii)	Carbon dioxide (as CO_2), percent by mass, <i>Max</i>	2.0	3.0	A-5
iv)	Fluoride (as F), percent by mass, <i>Max</i>	2.0	4.0	A-6
v)	Mixed aluminium and iron oxides (as Al_2O_3 and Fe_2O_3 respectively) percent by mass, <i>Max</i>	3.0	3.5	A-7
vi)	Moisture, percent by mass, <i>Max</i>	1.5	1.5	A-8
vii)	Magnesium oxide (as MgO), percent by mass, <i>Max</i>	0.5	0.5	A-9
viii)	Chloride (as Cl), percent by mass, <i>Max</i>	0.015	0.05	A-10
ix)	Organic matter and combined water, percent by mass, <i>Max</i>	2.0	1.5	A-11

4. PACKING AND MARKING

4.1 Packing — The material shall be supplied in bulk or in packages as agreed to between the purchaser and the supplier.

4.2 Marking — When supplied in packages, each package shall be securely closed and marked indelibly with the following information:

- a) Name and type of the material;
- b) Name of the supplier and his recognized trade-mark, if any;
- c) Gross and net mass;
- d) Date of packing; and
- e) Lot or batch number.

4.2.1 The packages may be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Definitions — For the purpose of this standard, the following definitions shall apply.

5.1.1 Lot — Quantity of the entire material which passes through half of the aperture size of the mesh corresponding to the declared particle size.

5.1.2 Average Mass of Particle — The average mass of a particle in a lot is the mass of a sphere whose diameter is equal to the average size of the lot.

5.1.3 Average Density — The average density of a particle in a lot is the weighted average of apatite and gangue.

5.2 Procedure

5.2.1 The run-of-mine (r.o.m.) for despatch to a consumer shall be broken in such a manner that 100 percent material passes through 125 mm IS sieve. Then proceed as per Table 2 which has been drawn on the basis that P 205 assay shall be within 1 percent of the time assay at 95 percent reliability.

TABLE 2 SAMPLING SCHEME

(Clause 5.2.1)

SL No.	PASSING THROUGH IS SIEVE	MINIMUM MASS OF SAMPLE	SAMPLING EQUIPMENT/PROCEDURE	REMARKS
i)	106-mm	4 000 kg	i) Scoop sampling of grand stock ii) Shovel sampling during unloading	Item (ii) should be preferred
ii)	22.4-mm	40 kg	Coning and quartering	—
iii)	2-mm	400 g	Coning and quartering	—

APPENDIX A

(Clause 3.2)

METHODS OF TEST FOR ROCK PHOSPHATE**A-1. QUALITY OF REAGENTS**

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PREPARATION OF TEST SAMPLE

A-2.1 Divide the sample by a riffle down to about 100 g. Grind it by mortar and pass it through 125 micron IS Sieve. Mix it well and store it in a moisture proof bottle.

A-3. TOTAL PHOSPHATES (QUINOLINE METHOD)

A-3.0 Two methods are prescribed for determining phosphates, namely, volumetric method and gravimetric method. In case of any dispute, volumetric method shall be taken as referee method.

A-3.1 Volumetric Method

A-3.1.0 Outline of the Method — The method is based on the precipitation of quinoline phosphomolybdate, which is dissolved in alkali solution and the excess of alkali is titrated against a standard acid. The main

*Specification for water for general laboratory use (*second revision*).

advantage over the ammonium phosphomolybdate method is that quinoline phosphomolybdate is less soluble and has a constant composition and quinoline is a sufficiently weak base which does not interfere in the titration.

A-3.1.1 Reagents

A-3.1.1.1 Quiniciac reagent — Dissolve 70 g of sodium molybdate in 150 ml of water. Dissolve 60 g of citric acid in mixture of 85 ml of nitric acid and 150 ml of water and cool. Gradually add molybdate solution to citric acid—nitric acid solution with stirring. Dissolve 5 ml of synthetic quinoline in a mixture of 35 ml of nitric acid and 100 ml of water. Gradually add this solution to molybdate citric acid—nitric acid solution; mix it and allow it to stand for 24 hours. Filter and add 280 ml of acetone, dilute it to one litre with water and mix. Store in a polyethylene bottle and keep in the dark.

A-3.1.1.2 Concentrated perchloric acid

A-3.1.1.3 Standard sodium hydroxide solution — 0.5 N.

A-3.1.1.4 Standard hydrochloric acid — 0.5 N.

A-3.1.1.5 Indicators

- a) *Thymol blue solution* — 0.1 percent. Add 2.2 ml of 0.1 N sodium hydroxide solution to 0.1 g of thymol blue and dilute to 100 ml with 50 percent alcohol.
- b) *Phenolphthalein* — 0.1 percent. Dissolve 0.1 g of the material in 100 ml of 50 percent alcohol.
- c) *Mixed indicator* — Mix three volume of (a) with two volume of (b).

A-3.1.2 Procedure

A-3.1.2.1 Weigh accurately about 1 g of the powdered and homogenized (see A-2) material into a 250-ml beaker, add 10 ml of water and 15 ml of aqua regia, warm until most of the sample dissolves, then evaporate to dryness. Cool and add 25 ml of perchloric acid and fume for 10 minutes under a fume hood. Allow to cool extract with 10 ml of hydrochloric acid (1 : 1) allowing the rinsing to run into the beaker. Transfer the contents of the beaker to the filter and wash with minimum volume of hot water till free from phosphate and make to 250 ml in a standard flask with water, mix the contents thoroughly.

A-3.1.2.2 Transfer 50 ml aliquot containing about 60 mg of phosphorus peroxide and 5 ml of concentrated nitric acid to 500 ml beaker, add 100 ml quiniciac reagent, immediately cover with watch glass, and

place on medium temperature hot plate. After solution comes to boiling point, move to cooler portion of hot plate and boil gently for 1 minute, let it cool until flask can be handled comfortably with bare hand.

A-3.1.2.3 Filter through a pulp pad employing suction and wash the flask, precipitate and filter with hydrochloric acid (1 : 9) three times followed by cold water until these are free from acid.

A-3.1.2.4 Transfer the precipitate along with the pulp into the beaker in which the precipitate was carried out. Add water to shake well. Add a measured excess of standard sodium hydroxide solution from a pipette to dissolve the precipitate. Add 1 ml of mixed indicator and titrate with standard hydrochloric acid solution to a grey-blue end point.

A-3.1.3 Calculation

$$\text{Total phosphates (as } P_2O_5 \text{) , } \frac{\text{percent by mass}}{\text{where}} = \frac{1.366 (AN_1 - BN_2)}{M}$$

where

A = volume in ml of standard solution of sodium hydroxide used;

N_1 = normality of standard solution of sodium hydroxide;

B = volume in ml of standard hydrochloric acid used to neutralize the excess alkali;

N_2 = normality of standard hydrochloric acid; and

M = mass in g of the material taken for test.

A-3.2 Gravimetric Method

A-3.2.0 *Outline of the Method* — The precipitate of quinoline phosphomolybdate is filtered through a sintered crucible. The crucible containing the precipitate is dried at 250°C to a constant mass.

A-3.2.1 *Procedure* — Filter the precipitate of quinoline phosphomolybdate through a sintered glass crucible of porosity No. 3, previously dried, cooled and weighed. Wash 5 times with 10 ml portions of water at room temperature. Dry it to constant mass at 250°C.

A-3.2.2 Calculation

$$\text{Total phosphates (as } P_2O_5 \text{) , } \frac{\text{percent by mass}}{\text{where}} = \frac{M_1 \times 3.21}{M_2}$$

where

M_1 = mass in g of the precipitate; and

M_2 = mass in g of the sample taken for the test.

A-4. SILICA

A-4.1 Apparatus

A-4.1.1 *Crucible Platinum* — 30 ml capacity.

A-4.1.2 *Muffle Furnace*

A-4.2 Reagents

A-4.2.1 *Concentrated Hydrochloric Acid* — see IS : 265 - 1976*.

A-4.2.2 *Boric Acid* — see IS : 10116 - 1982†.

A-4.2.3 *Perchloric Acid* — 65 percent.

A-4.2.4 *Dilute Sulphuric Acid* — 1 : 1 (*v/v*).

A-4.2.5 *Concentrated Hydrofluoric Acid*

A-4.3 Procedure — Weigh accurately 1 g of *prepared sample* (see A-2) into a 400-ml beaker. Add 1 g of boric acid and 10 to 15 ml water. Warm to dissolve boric acid. Cool slightly and add 20 ml of 70 percent perchloric acid. Evaporate to light fumes on moderate heat, cover and fume strongly for 15 minutes. Cool, add 1 ml of hydrochloric acid and 200 ml of water. Boil for 10 minutes, filter on a Whatman filter paper (No. 40) and wash thoroughly with hot water.

Place the paper with the precipitate in a platinum crucible, dry and ignite first at a low temperature until the carbon has been oxidized and finally at 1 050 to 1 100°C. Cool in a desiccator and weigh (M_2). Add 2 to 3 drops of sulphuric acid (1 : 1) and about 10 ml of hydrofluoric acid. Evaporate to fumes of sulphuric acid. Repeat evaporation with hydrofluoric acid to volatilize all silica. Fume off residual sulphuric acid over low flame and finally ignite at 1 050 to 1 100°C for 15 minutes. Dessicate, cool and weigh.

A-4.4 Calculation

$$\text{Silica (as SiO}_2\text{), percent by mass} = \frac{(M_2 - M_1) \times 100}{M}$$

where

M_1 = mass in g of the empty crucible;

M_2 = mass in g of the crucible and residue after ignition; and

M = mass in g of the sample taken for analysis.

*Specification for hydrochloric acid (*second revision*).

†Specification for boric acid.

NOTE — If the mass of residue after hydrofluoric acid treatment is more than 20 to 30 mg, then silica determination will have to be modified by fusion of insoluble residue with sodium carbonate, extraction with hydrochloric acid evaporation and baking of silica.

A-5. CARBON DIOXIDE

A-5.1 Outline of the Method — A known mass of the sample is made to react with an acid and the liberated carbon dioxide free from impurities is absorbed in previously weighed soda asbestos bulbs and weighed. From the difference in masses the percentage of carbon dioxide is calculated.

A-5.2 Apparatus — Apparatus consists of a 250-ml round bottom flask closed with rubber stopper or preferably standard glass joint with three holes for one tap funnel for addition of dilute hydrochloric acid, one water cooled reflux condenser and one tube for introduction of carbon dioxide free air. The top of the condenser is connected to a series of vessels B1 to B7 as follows:

B2, B3 and B4 — Nesbitt tubes (or Mid-vale tubes);

B1 and B7 — bubblers (Arnold bulb);

B5 — 125 ml bubbler; and

B6 — midvale tube.

The Arnold bulb (B1 and B7) contains syrupy phosphoric acid and are meant to indicate the rate of flow of gas through the apparatus (1 to 2 bubbles per second) and B5 bubbler contains concentrated sulphuric acid to remove bulk of moisture from the gas. The acid level in the bulbs B1, B5 and B7 should be just to cover the end of the delivery tube. Bulb B6 is meant for removal of carbon dioxide from the incoming air, packed with cotton wool to cover the end of the delivery tube and with soda asbestos to the level indicated in Fig. 1. B2(a) and B2(b) are packed with soda asbestos covered with 15 to 20 mm layer of anhydrous magnesium perchlorate B2(b) is meant for guard against any escape of Carbon dioxide from B2(a). B3 is for removal of hydrogen sulphide, packed with copper sulphate soaked in pumice stones or silica gel covered at the top with a 15 to 20 mm layer of anhydrous magnesium perchlorate. B4 contains magnesium perchlorate only. In each Nesbitt tube the reagent should be kept in place with the plug of cotton wool. The tubes are connected together with a suitable rubber or polyethylene tubing. The supply of air through the apparatus is obtained from a small filter pump and is regulated with a screw clip. A spirit lamp or micro-burner may be used to heat the flask.

A-5.2.1 Reagents

A-5.2.1.1 Dilute hydrochloric acid — 1 : 1 (v/v).

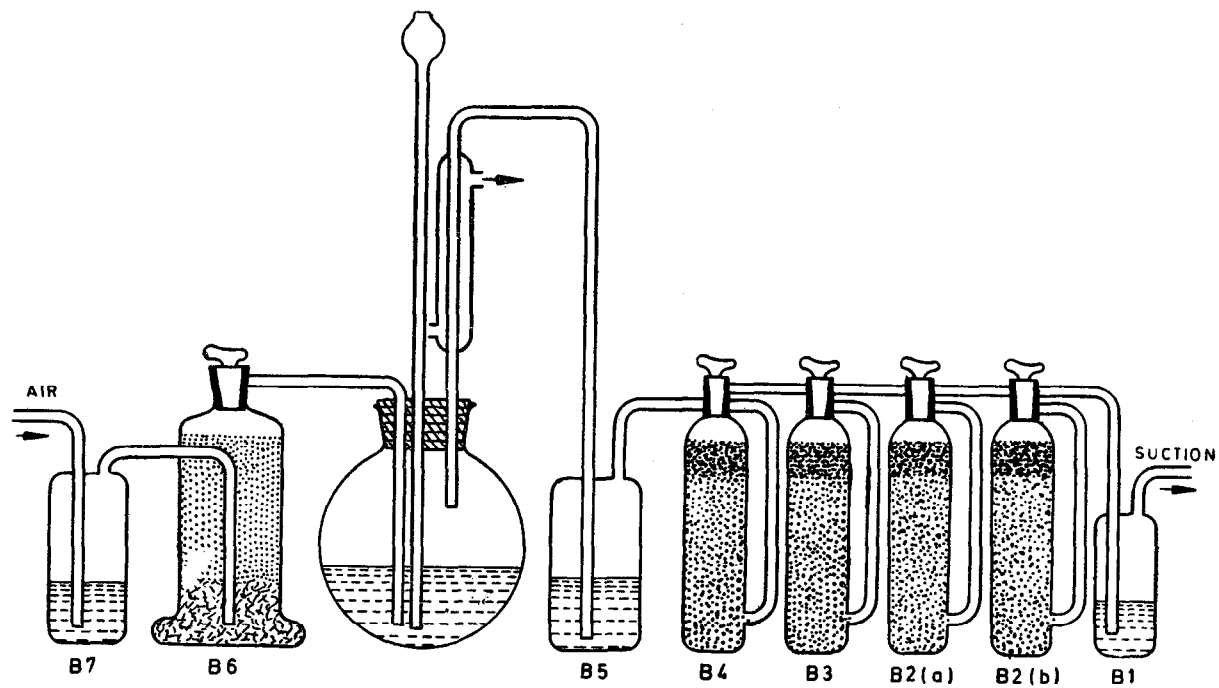


FIG. 1 APPARATUS FOR THE GRAVIMETRIC DETERMINATION OF CARBON DIOXIDE

A-5.2.1.2 *Magnesium perchlorate, anhydrous* — 1.18 to 7.10 micron IS sieve.

A-5.2.1.3 *Soda asbestos* — 2.00 mm to 1.18 mm IS sieve.

A-5.2.1.4 *Pumice impregnated with copper sulphate* — Anhydrous. Crush pumice to approximately 5 mm size, sift free from dust, and transfer 60 g of copper sulphate, evaporate to dryness while constantly stirring and then heat for 3 to 4 hours at 150 to 160°C in an air oven. Cool in a desiccator and preserve in a glass-stoppered bottle. Use freshly dried material in B3.

A-5.2.2 *Procedure* — Assemble the apparatus as shown in Fig. 1 with the bulbs packed as described. With all the taps fully open, start the pump pulling air through the apparatus and note that Arnold bulbs (B1 and B7) have air at about the same rate of 1 to 2 bubbles per second passing through them. Beginning at B7, close and then open each tap in turn and note that with each tap closed, the current of air through the Arnold bulb B1 ceases to flow. This will indicate that each tap is operating correctly. Now close all the taps and removing the Nesbitt tubes B2(a) and B2(b) to a balance case and allow to stand for 30 minutes. Carefully wipe the outside of the bulb with a clean cloth and weigh.

A-5.2.2.1 Weigh 1 g for grade 1 and 5 g for grade 2 of sample into the flask, moisten with a little water and transfer about 50 ml of dilute hydrochloric acid to the tap funnel. Reassemble the apparatus as shown in Fig. 1 with taps 1 to 7 open once more and with a clip between the suction pump and Arnold bulb B1 restricting the flow of air through the apparatus fully closed.

A-5.2.2.2 Start the pump and open the screw clip to produce a steady stream of bubbles through Arnold bulbs. Open the tap of the funnel to admit hydrochloric acid slowly to the flask. Close the tap of the funnel before all the acid has been added.

A-5.2.2.3 When all evolution of carbon dioxide has apparently ceased, boil the solution gently for about 10 minutes, turn off the burner and keep the air current flowing for a further period of 30 minutes. Close all the taps. Remove the Nesbitt tubes B2(a) and B2(b) to a balance case, allow to stand as before, wipe the tubes and weigh. Increase in mass is due to absorbed carbon dioxide. Run reagent blank through all the steps without taking the sample. This should be deducted from the mass of carbon dioxide determined above.

A-5.2.3 *Calculation*

$$\text{Carbon dioxide, percent by mass} = \frac{(M_1 - B_1) \times 100}{M}$$

where

M_1 = mass in g of Nesbitt tubes B2(a) and B2(b) after the test;

M = mass in g of the material taken for the test; and

B_1 = mass in g of Nesbitt tube B2(a) and B2(b) for blank experiment.

A-6. DETERMINATION OF FLUORINE BY DISTILLATION METHOD

A-6.1 Outline of the Method — Fluorine is separated as hydrofluosilicic acid by distillation with perchloric acid and determined by titration against standard thorium nitrate solution using sodium alizarine sulphonate as indicator.

A-6.2 Apparatus

A-6.2.1 The distillation apparatus (*see* Fig. 2) consist of a 250-ml distillation flask (A), side arm of which is connected to a condenser (B). The neck of the flask is fitted with a two-hold rubber-stoppered flask to a steam generator (E) which is equipped with two discharges, one tube (F) to release steam and the other (D) to deliver steam into the distillation flask (A). The distillate is collected in a conical flask (G).

A-6.3 Reagents

A-6.3.1 *Perchloric Acid* — 70 percent.

A-6.3.2 *Sodium Alizarine Sulphonate Indicator* — Dissolve 0.05 g of alizarine sulphonate in 100 ml of water, and store in a dropping bottle.

A-6.3.3 *Sodium Hydroxide Solution* — Dissolve 10 g of sodium hydroxide pellets in water, cool and dilute to one litre. Mix well.

A-6.3.4 *Dilute Hydrochloric Acid* — Dilute 19.6 ml of concentrated hydrochloric acid to 1 000 ml with water. Mix well.

A-6.3.5 *Buffer Solution* — Dissolve 9.448 g of monochloroacetic acid in 100 ml of water. Add 50 ml of sodium hydroxide (1N). Check the pH by pH meter. The pH should be 3.0 ± 0.1 .

A-6.3.6 *Standard Thorium Nitrate Solution* — 0.025 M. Dissolve 13.806 g of thorium nitrate in water and make up to one litre. Mix well. Standardized against sodium fluoride solution (*see* 5.3.7) and carry out all the stages of procedure given in 5.4.3. Preferably a calibration curve may be prepared showing volume of thorium nitrate titre against quantity of sodium fluoride.

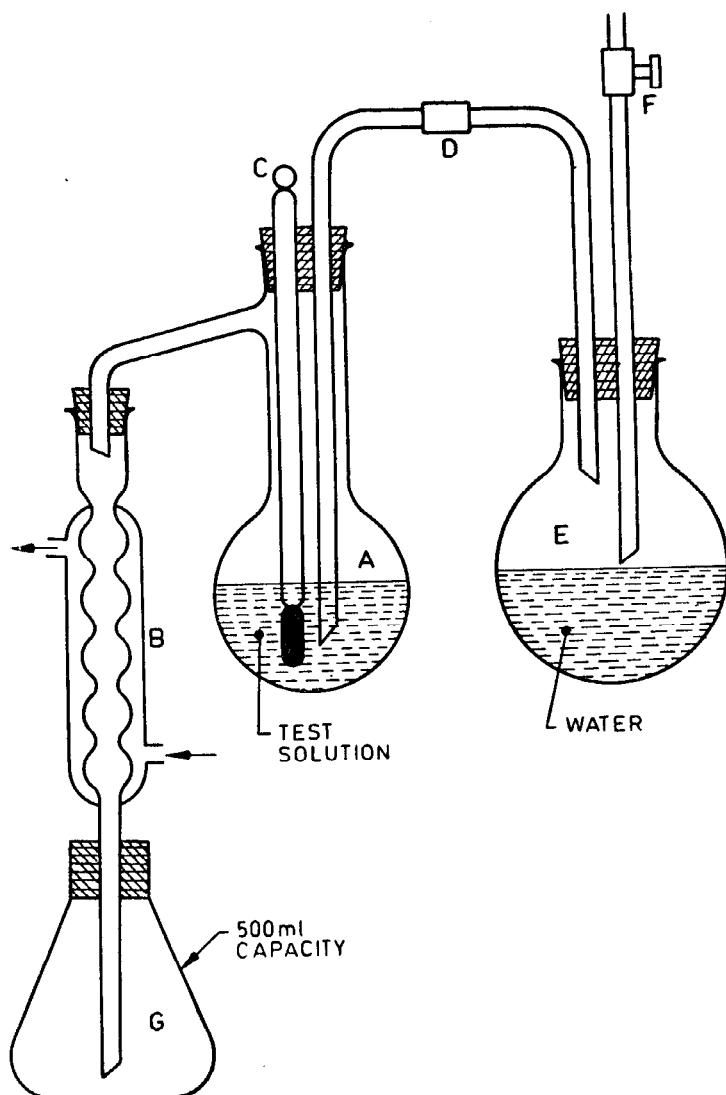


FIG. 2 APPARATUS FOR DETERMINATION OF FLUORINE

A-6.3.7 Standard Sodium Fluoride Solution — Dissolve 2.211 g of sodium fluoride (analytical reagent grade), previously dried at 120°C to constant mass, in one litre of water in a volumetric flask. One millilitre of this solution is equal to one milligram of fluorine.

A-6.4 Procedure

A-6.4.1 Transfer 0.5 g of an accurately weighed sample to a 250-ml distillation flask containing 0.5 g of sodium silicate and a few glass beads. Add 10 ml of perchloric acid (phosphoric acid may be used along with perchloric acid as it helps in releasing fluorine for the complex).

A-6.4.2 Place a 1 000-ml flask under the condenser. Connect the apparatus as shown in Fig. 1, distillation flask (A) to the condenser and distil the solution until the boiling point reaches 140°C. While this is being done, heat the water in the steam generator (E) to boiling but do not allow the steam to get in the flask (A). When the temperature reaches 140°C, admit steam into the flask (A). Maintain the temperature $140 \pm 2^\circ\text{C}$ by adjusting the steam flow and heating of flask (A). Distil at such a rate that no fumes or mist escape from the volumetric flask. Continue distillation until nearly 250 ml are collected in a 100-ml flask, containing 25 ml of sodium hydroxide solution.

A-6.4.3 Transfer to a 250-ml volumetric flask. Make the volume up to the mark and mix well. Pipette out 50 ml aliquot into a 250-ml beaker. Add 50 ml of water, 10 drops of sodium alizarine sulphonate indicator solution and mix. Add sodium hydroxide solution slowly with stirring till the solution becomes pink. Add dilute hydrochloric acid drop by drop with stirring till the pink colour is just discharged leaving the solution just acid. Add two millilitres of buffer solution and titrate till the permanent pink end point is reached. The colour intensity at the end point should be kept in mind and the titration should be stopped exactly at the same colour intensity for both the standard thorium nitrate solution (see A-6.3.6) and the test solution titration.

A-6.5 Calculation

$$\text{Fluoride (as F), percent by mass} = \frac{A \times B}{C} \times 100$$

where

A = volume in ml of standard thorium nitrate solution;

B = fluorine equivalent of the standard thorium nitrate solution in g/ml; and

C = mass in g of the sample represented by the aliquot taken.

A-7. MIXED OXIDES

A-7. Aluminium oxide is estimated first by the gravimetric method removing the phosphates. This value is then added to the value of iron oxide determined in **A-7.2** to get the value of mixed oxides.

A-7.1.0 Outline of the Method — The aluminium is precipitated in presence of acetic acid by boiling the solution with diammonium phosphate and sodium thiosulphate. The precipitate is ignited and weighed as aluminium phosphate.

A-7.1.1 Reagent

A-7.1.1.1 Sodium thiosulphate solution — 20 percent (m/v).

A-7.1.1.2 Diammonium phosphate solution — 10 percent (m/v).

A-7.1.1.3 Dilute hydrochloric acid — 1 : 3 (v/v).

A-7.1.1.4 Acetic-acetate buffer solution — Dissolve 77 g of ammonium acetate in 600 ml of water and add 330 ml of glacial acetic acid. Dilute to one litre with water.

A-7.1.1.5 Ammonium hydroxide solution

A-7.1.1.6 Methyl orange indicator solution — Dissolve 0.1 g of methyl orange in 20 percent rectified spirit.

A-7.1.2 Procedure

A-7.1.2.1 Weigh accurately about 1 g of the *prepared sample* (see **A-2**) to a 500-ml wide mouth flask, add 30 to 40 ml of concentrated hydrochloric acid and evaporate to dryness. Bake for a few minutes, cool and add 5 ml of concentrated hydrochloric acid. Boil until the brown fumes have been expelled and 50 ml of water, cool to room temperature, filter and make to 500 ml in standard flask.

A-7.1.2.2 Transfer an aliquot of the stock solution (**A-6.2.1**) containing up to 50 mg of aluminium oxide in a 500-ml beaker. Add 200 ml of water, 30 ml of diammonium phosphate solution and 2 drops methyl orange indicator. Neutralize with ammonium hydroxide and then acidify by adding exactly 2 ml of hydrochloric acid solution.

A-7.1.2.3 Add 50 ml of sodium thiosulphate solution and a small amount of macerated ashless filter paper and heat until it starts boiling and add about 25 ml of acetate-acetic acid buffer and boil for 10 minutes or more in order to coagulate the precipitated sulphur and to reduce the iron to the ferrous condition. Ferrous phosphate is not precipitated in presence of acetic acid. Allow the precipitate to settle and filter. Wash with

cold water until free from chlorides, ignited at 800°C and weigh as aluminium phosphate and calculate the percentage of aluminium oxide.

A-7.1.3 Calculation

Aluminium oxide (as Al_2O_3), percent by mass

$$= \frac{M_1 \times 0.418 \times 100}{M_2} \times \text{dilution factor}$$

where

M_1 = mass in g of the precipitate; and

M_2 = mass in g of the material taken for the test.

A-7.2 Iron Oxide

A-7.2.1 Apparatus

A-7.2.1.1 Photoelectric colorimeter

A-7.2.2 Reagents

A-7.2.2.1 Citric acid solution — 20 percent (*m/v*).

A-7.2.2.2 Thioglycolic acid

A-7.2.2.3 Dilute ammonia solution — 10 percent (*m/v*).

A-7.2.2.4 Standard iron solution (stock solution) — Dissolve 0.35 g analytical grade iron or powdered electrolytic iron in 10 ml of sulphuric acids 1 : 10 (*v/v*), and make up to one litre, or dissolve 2.456 g of analytical grade ferrous ammonium sulphate in 300 ml of water containing 5 ml concentrated sulphuric acid. Make up volume to one litre with water in a volumetric flask.

A-7.2.2.5 Working standard — Dilute 20 ml of standard iron solution (**A-7.2.2.4**) to one litre in a volumetric flask. One ml of the solution contains 10 μg of iron oxide.

A-7.2.3 Procedure — Place 0, 5, 10, 20, 30, 40 and 50 ml of the iron solution into 100 ml volumetric flasks and dilute to about 80 ml with water. Add 4 ml citric acid solution and 0.2 ml of thioglycolic acid to each flask and shake. Render alkaline with dilute ammonia solution. Shake again and make up to mark with water. Mix and allow to stand for 5 minutes. Measure optical density of each solution against reagent blank at 5330 Å (filter 530 nm or green filter) using 4 cm cells (or test tube 12 to 18 mm)

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subtract blank optical density. Plot optical density against μg of iron oxide in 100 ml solution.

A-7.2.3.1 Weigh accurately 1 g of the prepared sample to 500-ml wide mouth flask, add 30 to 40 ml of concentrated hydrochloric acid containing 3 to 4 ml of nitric acid and evaporate to dryness. Bake for a few minutes, cool and add 5 ml of concentrated hydrochloric acid. Boil until the brown fumes have been expelled, add 50 ml of water, cool to room temperature, filter and dilute to 500 ml in a standard flask.

A-7.2.3.2 Pipette suitable aliquot (equivalent to 200 to 250 μg of iron oxide from the prepared solution (**A-7.2.3.1**) into a 100-ml volumetric flask and dilute to about 80 ml with water. Add 4 ml of citric acid solution and 0.2 ml of thioglycolic acid. Shake, render alkaline with dilute ammonia solution. Shake again and make up to the mark with water, mix and allow to stand for 5 minutes. Read optical density at 5330 Å against water as done in calibration.

Determine a blank in exactly the same way and subtract the blank optical density from the sample optical density. Refer to the calibration graph and get the equivalent μg of iron oxide.

A-7.2.4 Calculation

$$\text{Iron Oxide (as Fe}_2\text{O}_3 \text{), } \frac{\mu\text{g of iron oxide in the aliquot}}{\text{percent by mass}} = \frac{20 V}{V}$$

where

V = volume of aliquot taken in ml from the prepared solution.

A-7.3 Calculation

$$\begin{array}{lcl} \text{Mixed oxides (iron oxide} & & \text{Iron oxide (A-7.2.4) +} \\ \text{and aluminium oxide),} & = & \text{Aluminium oxide} \\ \text{percent by mass} & & \text{(A-7.1.3)} \end{array}$$

A-8. MOISTURE

A-8.1 Procedure — Take about 10 g of the powdered sample passing through 180 micron IS sieve on a tared petri dish. Spread it over uniformly and weigh. Heat the petri dish and the material in an oven heated at 105 to 110°C for one hour. Cool the petri dish and the material in desiccator and weigh. Repeat this operation till a constant mass is obtained.

A-8.2 Calculation

$$\text{Moisture, percent by mass} = \frac{M_2 - M_1}{M} \times 100$$

where

M_2 = mass in g of the petri dish and sample before drying;

M_1 = mass in g of the petri dish and sample after drying; and

M = mass in g of the material taken for the test.

A-9. MAGNESIUM OXIDE**A-9.1 Reagents**

A-9.1.1 *Citric Acid Crystals*

A-9.1.2 *Diammonium Phosphate* — 10 percent (*m/v*).

A-9.1.3 *Concentrated Hydrochloric Acid* — See IS : 276-1976*.

A-9.1.4 *Dilute Nitric Acid* — 50 percent.

A-9.1.5 *Dilute Sulphuric Acid* — 50 percent.

A-9.1.6 *Ammonium Hydroxide Solution*

A-9.2 Procedure

A-9.2.1 Take 2 g of sample in a 400-ml beaker, add 30 ml concentrated hydrochloric acid and digest it on the hot plate to get a thick syrupy liquid. Again add 10 ml concentrated hydrochloric acid and evaporate to syrupy liquid. (Avoid baking and drying the acid). Then add 50 ml dilute nitric acid and heat it on a hot plate till white fumes. Add about 150 ml of water, boil it for 5 minutes, cool and filter through Whatman Filter Paper (No. 41) into a 250-ml volumetric flask and wash the residue and make up to 250 ml with water.

A-9.2.2 Pipette out 50 ml aliquot in a 400-ml beaker, add 50 ml water and 10 ml dilute sulphuric acid. Add excess methanol to separate out calcium sulphate. Stir well and allow to stand for one hour. Filter through Whatman Filter Paper (No. 41), wash the precipitate at least 5 times by methanol (Filtrate should be clear). Evaporate the filtrate to near about 50 ml solution. Cool the solution and add 5 to 7 g of citric acid crystals and 30 ml of diammonium phosphate. Make the solution alkaline to litmus paper by adding ammonia solution. Add 10 to 20 ml excess ammonia, stir well and then allow the precipitate of magnesium pyrophosphate to settle at least for 6 hours (preferably overnight). Filter through Whatman Filter

*Specification for hydrochloric acid (*second revision*).

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paper No. 40, wash it with 5 percent ammonia solution. Ignite the residue in a weighed platinum crucible and weigh it after cooling.

A-9.3 Calculation

$$\text{Magnesium oxide (as MgO), percent by mass} = \frac{M_1 \times 181.05}{M}$$

where

M_1 = mass in g of the residue (magnesium pyrophosphate);
and

M = mass in g of the sample taken for the test.

A-10. CHLORIDE

A-10.1 Reagents

A-10.1.1 Standard Sodium Chloride Solution — Dissolve 1.648 g of dried sodium chloride in water and make up to 1 litre. One ml of this solution is equivalent to 0.1 mg of chlorine.

A-10.1.2 Silver Nitrate Solution — 1 percent.

A-10.2 Procedure

A-10.2.1 Transfer 2 g of sample in a beaker with 50 ml water and 2 ml concentrated nitric acid. Digest it for 15 minutes. Cool it and make up the solution to 100 ml.

A-10.2.2 Filter and pipette out 10 ml aliquot in 50 ml wide-mouth Nessler cylinder. Add 2 ml of silver nitrate. Prepare a blank by adding silver nitrate and a drop of concentrated nitric acid in another Nessler cylinder. Add standard chloride solution and make the turbidity as equal to sample.

A-10.3 Calculation

$$\text{Chloride (as Cl), percent by mass} = \frac{V \times \text{dilution factor}}{M}$$

where

V = volume in ml of standard sodium chloride solution required; and

M = mass in g of the sample taken for test.

A-11. ORGANIC MATTER

A-11.0 Outline of the Method — Organic matter is determined from the following expression:

$$\text{Organic matter, percent by mass} = [\text{Loss on Ignition} - (\text{Carbon-dioxide}) \quad \text{Loss at } 230^\circ\text{C}]$$

A-11.1 Loss on Ignition

A-11.1.1 Ignite about 2 g of the sample in a platinum crucible at about 950°C on a burner for 2 hours, then cool it and weigh it again. Determine the loss in mass after ignition.

A-11.1.2 Calculation

$$\text{Loss on ignition, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g after ignition; and

M_2 = mass in g of the sample taken for the test.

A-11.2 Loss at 230°C

A-11.2.1 Weigh about 5 g (M) of the sample in a previously dried and weighed moisture dish (M_1). Dry it in an oven at 230°C for one and half hour, cool it in a desiccator and weigh it again (M_2).

A-11.2.2 Calculation

$$\text{Loss at 230°C, percent by mass} = \frac{(M_1 - M_2)}{M} \times 100$$

A-11.3 Carbon Dioxide

A-11.3.1 To be determined as given in clause A-5.

A-11.4 Calculation

$$\begin{aligned} \text{Organic matter, percent by mass} = & [\text{Loss on ignition (A-11.1.2)} \\ & - \text{Carbon dioxide (A-5.2.3)} \\ & + \text{Loss at 230°C (A-11.2.2)}]. \end{aligned}$$

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>	<i>Definition</i>
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²